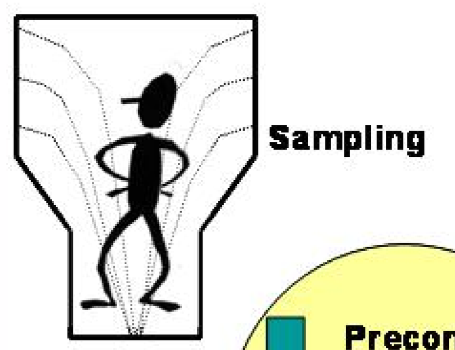
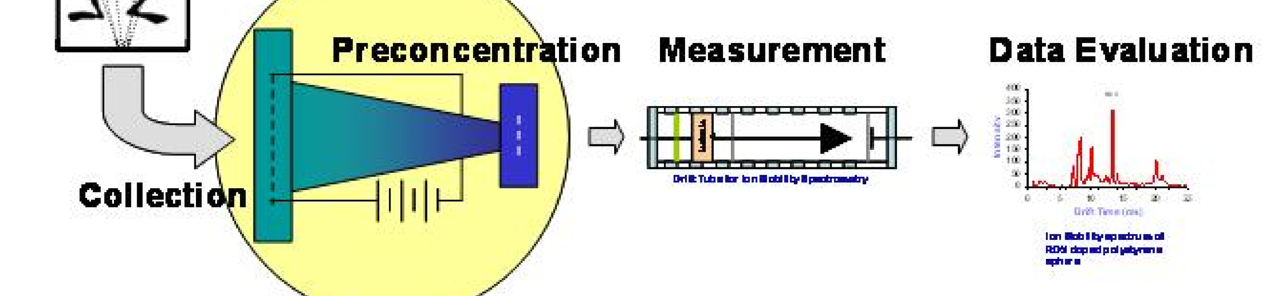


The Role of Preconcentration in the Detection of Explosive Particles



Vapor Preconcentration During Explosive Detection: Study of Variables in a NIST Standard Test System

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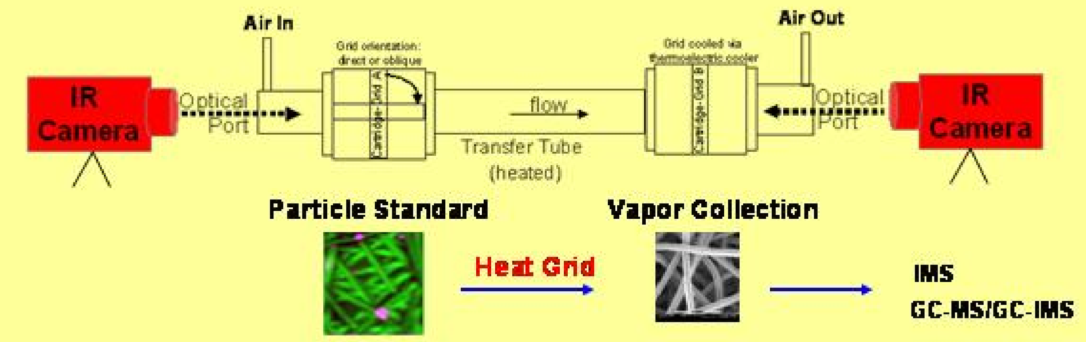
Purpose: To establish a standard testing system for the study and optimization of explosive detection systems, especially in regard to the transport and preconcentration of particles and vapors.

Background: Detection technology for explosives and other banned substances often rely on the chemical signals from particles. When these particles are suspended in the atmosphere, usually at ultratrace levels, their chemical signals must be enhanced at least to the point where detection technologies are capable of achieving adequate levels of sensitivity and selectivity for accurate responses. This signal enhancement may be realized through particle preconcentration; i.e., collecting explosive particles from a large volume of air by selective filtration, and processing the captured particles to a form detectable by current technologies. To collect explosive particles in portals, air samples are filtered through large metal grids, which are subsequently heated to vaporize the compounds. These compounds are recollected onto a small filter suitable for analysis. While current explosive screening systems have been successful, inherent in all measurement systems are a large number of process and analytical variables that must be understood in order to design appropriate and reliable methods that can reach the required analytical goals under statistical control. Currently, there is insufficient fundamental information for this purpose available to system designers, instrument manufacturers, and public safety officials, and standards do not yet exist on which to compare capabilities across technologies, to base accreditations, to limit liability, and to foster public confidence in the reliability of screening methods.

Stage 1: Design and Fabrication



Preconcentration system – direct transfer configuration

View of direct transfer module, cartridge, and Grid A

Vapor collection module, showing Grid B

Vapor collection module, showing PTFE mask removed from Grid B and thermoelectric cooler

Grid A – camera view through BaF_2 window

Infra-red camera

Grid B – camera view through BaF_2 window

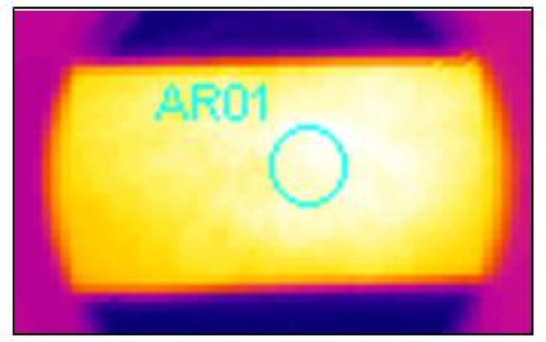
Preconcentration System – oblique transfer configuration

Five views of oblique transfer module. First image shows camera view flange. In other images this flange has been removed, the module rotated onto that face, and the cartridge access plate removed. The second & third images show the black cartridge cover, the third & fourth images show Grid A in the cartridge, and the fifth image reveals the camera view port upon removal of the cartridge.

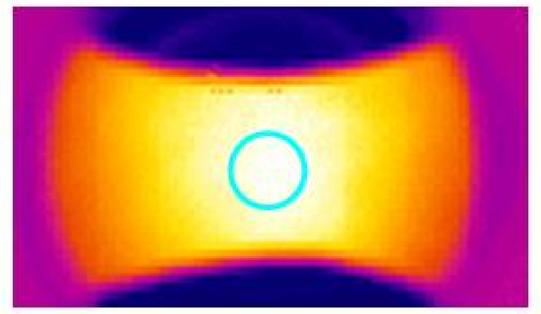
Stage 2: Characterization of Grid A Prototypes by IR Thermometry

Purpose: To identify a small area on grid where temperature may be precisely controlled during flash vaporization of explosive samples

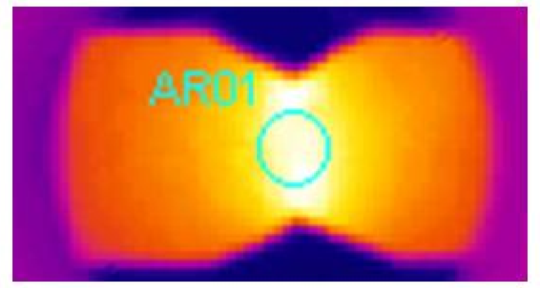
Effect of Grid Shape on Temperature Distribution During 6 sec, 20 amp Burst



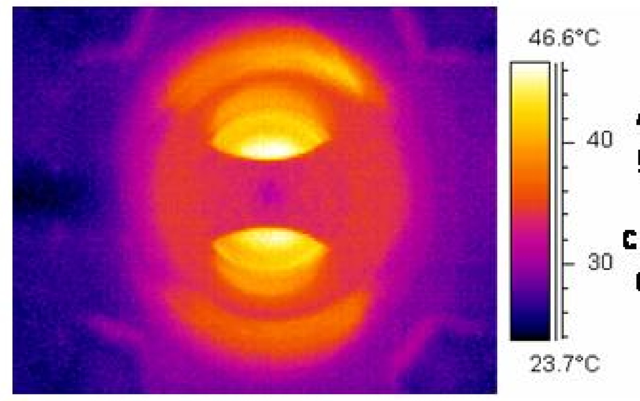
Rectangular Grid
Resistance = 0.1 ohm
s = 4.6 °C in circle
Note variation across grid
Tmax off-center



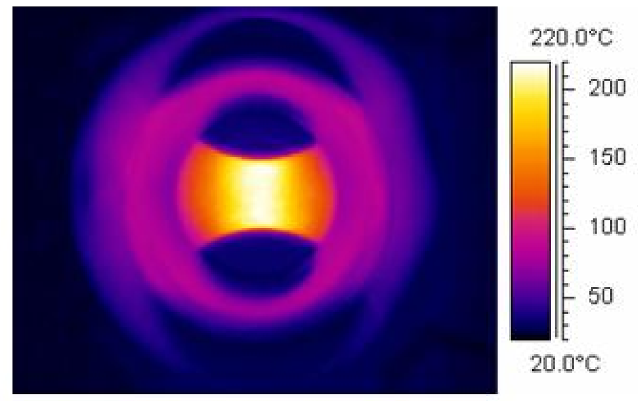
Concave Grid (design of choice)
Resistance = 0.1 ohm
s = 3.0 °C in circle – ***lowest variation***



Notched Grid
Resistance = 0.1 ohm
s = 6.1 °C in circle



After a sample is applied to the grid, evaporation of the solvent causes initial cooling at the center of the grid. When the spot disappears the sample is ready for flash vaporization.



The sample is vaporized by resistively heating the grid.

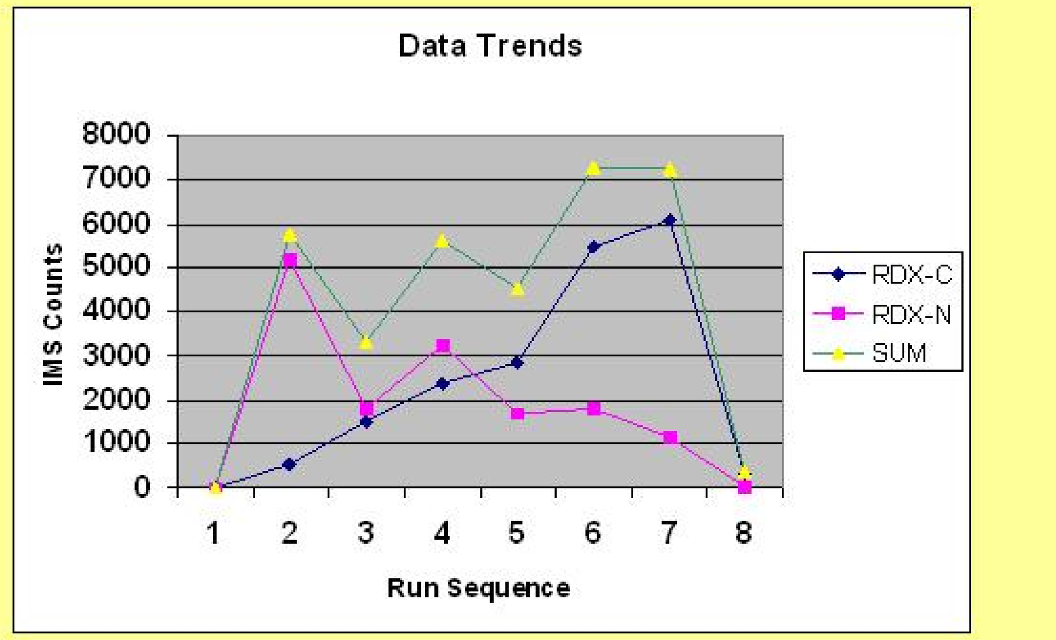
Stage 3: Exploration of Test System by Ion Mobility Spectrometry

Table 1. IMS Data from Initial Tests - Proof of Concept

Run	Sample	Flash Time (s)	RDX-C counts	RDX-N counts	Comments
1	Process blank	6	ND	ND	
2	10 ng RDX Grid A	6	546	5206	12572 NO3
3	10 ng RDX Grid A	10	1510	1801	Sample off-center
std	5 ng RDX Grid B	-	14085	2709	
4	10 ng RDX Grid A	6	2375	3247	
5	10 ng RDX Grid A	8	2840	1685	
std	1 ng RDX Grid B	-	4587	500	
6	10 ng RDX Grid A	10	5493	1786	Edges tested clean
7	10 ng RDX Grid A	6	6098	1140	
std	1 ng RDX Grid B	-	2566	137	
8	Process blank	10	334	28	
std	5 ng RDX Grid B	-	15697	4907	

Test system operated with variables at Level 1 (see Table 2)

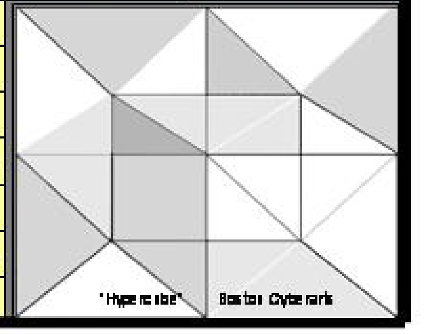
IMS data taken on a Barringer (Smiths Detection) IonScan 500



Stage 4: (Pending) Characterizations by Designed Factorial Experiments

Table 2. Significant Variables and Levels

Code	Category	Variable	Level 1	Level 2
A1	Gas	Type	Air	Helium
A2	Gas	Flow	5 lpm	10 lpm
B1	Configuration	Grid A	Direct	Oblique
B2	Configuration	Transfer Diameter	2.5 cm	1.0 cm
B3	Configuration	Transfer Length	0.3 m	1.0 m
B4	Configuration	Transfer Path	Straight	90° Bend
C1	Grid A	Metal Type	SS316L	Ni
C2	Grid A	Fiber Diameter	22 µm	33 µm
C3	Grid A	Grid Thickness	0.7 mm	0.5 mm
D1	Grid B	Metal Type	SS 316L	Ni
D2	Grid B	Fiber Diameter	22 µm	33 µm
D3	Grid B	Grid Thickness	0.7 mm	0.4 mm
D4	Grid B	Grid Porosity	89 %	70 %
D5	Grid B	Mask Diameter	1.6 cm	1.0 cm
E1	Initial Temperature	Module D/O	30 °C	100 °C
E2	Initial Temperature	Module T	80 °C	150 °C
E3	Initial Temperature	Module C	50 °C	20 °C
E4	Initial Temperature	Grid A	30 °C	50 °C
E5	Initial Temperature	Grid B	40 °C	20 °C
F1	Sample	Chemical	RDX	TNT
F2	Sample	Particle Diameter	From soln	6 µm
F3	Sample	Amount	10 ng	5 ng
G1	Activation	Amperage	20 A	15 A
G2	Activation	Time	6 s	12 s
Response Variables				
X1	Grid A Temperature	Slope		
X2	Grid A Temperature	Peak		
X3	Grid A Temperature	Standard Deviation		
Y1	Grid B Temperature	Delta		
Y2	Grid B Temperature	Standard Deviation		
Z1	Sample – Grid B	Amount		



- **Problem: 2-level full factorial design for 24 process variables calls for 16,777,216 experimental observations**
- **Approach: Pilot study to prioritize variables and identify their interactions, then subsequent focused studies to help determine answers to specific questions.**
  - **Pilot: 2-level fractional factorial design: 2**(24-17) = 128 observations**
  - **Focused study using significant variables in fractional factorial design**
    - **Example Question: Does gas type and flow influence yield on grid B materials for any explosive compound?**
    - **Obtain observations of Z1 for each explosive compound (F1) across most pertinent variables identified in pilot (e.g. A1, A2, D1, D4, E5, G1, G2) in 2**(7-2) fractional factorial design → 32 observations per explosive (caveat: main effects would be confounded with any 2-factor interactions)**

Two-level fractional factorial design for 7 variables

Obs	A1	A2	D1	D4	E5	G1	G2
1	-1	-1	-1	-1	-1	-1	+1
2	+1	-1	-1	-1	+1	+1	-1
3	-1	+1	-1	-1	+1	-1	-1
⋮	⋮	⋮	⋮	⋮	⋮	⋮	⋮
30	+1	+1	+1	-1	-1	-1	-1
31	-1	-1	+1	+1	+1	-1	-1
32	+1	+1	+1	+1	+1	+1	+1

NIST
National Institute of Standards and Technology
Technology Administration
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